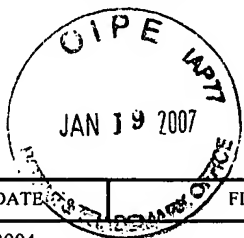




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Herbert B. Keil  
KEIL & WEINKAUF  
1350 Connecticut Ave., N.W.  
Washington, DC 20036

EXAMINER

BOYER, RANDY

ART UNIT PAPER NUMBER

1764

SHORTENED STATUTORY PERIOD OF RESPONSE	MAIL DATE	DELIVERY MODE
3 MONTHS	01/09/2007	PAPER

Please find below and/or attached an Office communication concerning this application or proceeding.

If NO period for reply is specified above, the maximum statutory period will apply and will expire 6 MONTHS from the mailing date of this communication.

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<b>Office Action Summary</b>	<b>Application No.</b> 10/806,191	<b>Applicant(s)</b> BARTENBACH ET AL.	
	<b>Examiner</b> Randy Boyer	<b>Art Unit</b> 1764	

**-- The MAILING DATE of this communication appears on the cover sheet with the correspondence address --**

**Period for Reply**

A SHORTENED STATUTORY PERIOD FOR REPLY IS SET TO EXPIRE 3 MONTH(S) OR THIRTY (30) DAYS, WHICHEVER IS LONGER, FROM THE MAILING DATE OF THIS COMMUNICATION.

- Extensions of time may be available under the provisions of 37 CFR 1.136(a). In no event, however, may a reply be timely filed after SIX (6) MONTHS from the mailing date of this communication.
- If NO period for reply is specified above, the maximum statutory period will apply and will expire SIX (6) MONTHS from the mailing date of this communication.
- Failure to reply within the set or extended period for reply will, by statute, cause the application to become ABANDONED (35 U.S.C. § 133). Any reply received by the Office later than three months after the mailing date of this communication, even if timely filed, may reduce any earned patent term adjustment. See 37 CFR 1.704(b).

**Status**

- 1) ☒ Responsive to communication(s) filed on 23 March 2004.
- 2a) ☐ This action is **FINAL**.                      2b) ☒ This action is non-final.
- 3) ☐ Since this application is in condition for allowance except for formal matters, prosecution as to the merits is closed in accordance with the practice under *Ex parte Quayle*, 1935 C.D. 11, 453 O.G. 213.

**Disposition of Claims**

- 4) ☒ Claim(s) 1-20 is/are pending in the application.
- 4a) Of the above claim(s) \_\_\_\_\_ is/are withdrawn from consideration.
- 5) ☐ Claim(s) \_\_\_\_\_ is/are allowed.
- 6) ☒ Claim(s) 1-20 is/are rejected.
- 7) ☒ Claim(s) 6 is/are objected to.
- 8) ☐ Claim(s) \_\_\_\_\_ are subject to restriction and/or election requirement.

**Application Papers**

- 9) ☐ The specification is objected to by the Examiner.
- 10) ☐ The drawing(s) filed on \_\_\_\_\_ is/are: a) ☐ accepted or b) ☐ objected to by the Examiner.  
Applicant may not request that any objection to the drawing(s) be held in abeyance. See 37 CFR 1.85(a).  
Replacement drawing sheet(s) including the correction is required if the drawing(s) is objected to. See 37 CFR 1.121(d).
- 11) ☐ The oath or declaration is objected to by the Examiner. Note the attached Office Action or form PTO-152.

**Priority under 35 U.S.C. § 119**

- 12) ☒ Acknowledgment is made of a claim for foreign priority under 35 U.S.C. § 119(a)-(d) or (f).
- a) ☒ All    b) ☐ Some \* c) ☐ None of:
1. ☒ Certified copies of the priority documents have been received.
  2. ☐ Certified copies of the priority documents have been received in Application No. \_\_\_\_\_.
  3. ☐ Copies of the certified copies of the priority documents have been received in this National Stage application from the International Bureau (PCT Rule 17.2(a)).

\* See the attached detailed Office action for a list of the certified copies not received.

**Attachment(s)**

- |  |   |
|--|---|
| 1) <input checked="" type="checkbox"/> Notice of References Cited (PTO-892)  | 4) <input type="checkbox"/> Interview Summary (PTO-413)<br>Paper No(s)/Mail Date. _____ |
| 2) <input type="checkbox"/> Notice of Draftsperson's Patent Drawing Review (PTO-948)   | 5) <input type="checkbox"/> Notice of Informal Patent Application                       |
| 3) <input checked="" type="checkbox"/> Information Disclosure Statement(s) (PTO/SB/08)<br>Paper No(s)/Mail Date <u>23 March 2004</u> . | 6) <input type="checkbox"/> Other: _____  |

## **DETAILED ACTION**

### ***Claim Objections***

1. Claim 6 is objected to for improper use of the plural form. As submitted, claim 6 reads "A process as claimed in claims 1, . . .". Examiner suggests correction by amending the claim to read "A process as claimed in claim 1, . . .". Appropriate correction is required.

### ***Claim Rejections - 35 USC § 112***

2. The following is a quotation of the second paragraph of 35 U.S.C. 112:

The specification shall conclude with one or more claims particularly pointing out and distinctly claiming the subject matter which the applicant regards as his invention.

3. 35 U.S.C. 101 reads as follows:

Whoever invents or discovers any new and useful process, machine, manufacture, or composition of matter, or any new and useful improvement thereof, may obtain a patent therefor, subject to the conditions and requirements of this title.

4. Claim 8 is rejected under 35 U.S.C. 112, second paragraph, as being indefinite for failing to particularly point out and distinctly claim the subject matter which applicant regards as the invention.
5. Claim 8 includes the limitation "a fire-resistant ceramic stable at reaction temperature having an alumina content of at least 80%." The claim language is indefinite since it does not provide the basis for which the alumina content is to be measured (e.g. 80 wt. %, 80 mol %, etc.).

Art Unit: 1764

6. Claim 20 is rejected under 35 U.S.C. 112, second paragraph, as being indefinite for failing to particularly point out and distinctly claim the subject matter which applicant regards as the invention.

7. Claim 20 provides for the use of "a process as claimed in claim 1" or "a reactor as claimed in claim 8", but, since claims 1 and 8 do not set forth any steps involved in the method/process, it is unclear what method/process applicant is intending to encompass. A claim is indefinite where it merely recites a use without any active, positive steps delimiting how this use is actually practiced.

Claim 20 is rejected under 35 U.S.C. 101 because the claimed recitation of a use, without setting forth any steps involved in the process, results in an improper definition of a process, i.e., results in a claim which is not a proper process claim under 35 U.S.C. 101. See for example *Ex parte Dunki*, 153 USPQ 678 (Bd.App. 1967) and *Clinical Products, Ltd. v. Brenner*, 255 F. Supp. 131, 149 USPQ 475 (D.D.C. 1966).

### ***Claim Rejections - 35 USC § 103***

8. The following is a quotation of 35 U.S.C. 103(a) which forms the basis for all obviousness rejections set forth in this Office action:

(a) A patent may not be obtained though the invention is not identically disclosed or described as set forth in section 102 of this title, if the differences between the subject matter sought to be patented and the prior art are such that the subject matter as a whole would have been obvious at the time the invention was made to a person having ordinary skill in the art to which said subject matter pertains. Patentability shall not be negated by the manner in which the invention was made.

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9. The factual inquiries set forth in *Graham v. John Deere Co.*, 383 U.S. 1, 148 USPQ 459 (1966), that are applied for establishing a background for determining obviousness under 35 U.S.C. 103(a) are summarized as follows:

1. Determining the scope and contents of the prior art.
2. Ascertaining the differences between the prior art and the claims at issue.
3. Resolving the level of ordinary skill in the pertinent art.
4. Considering objective evidence present in the application indicating obviousness or nonobviousness.

10. This application currently names joint inventors. In considering patentability of the claims under 35 U.S.C. 103(a), the examiner presumes that the subject matter of the various claims was commonly owned at the time any inventions covered therein were made absent any evidence to the contrary. Applicant is advised of the obligation under 37 CFR 1.56 to point out the inventor and invention dates of each claim that was not commonly owned at the time a later invention was made in order for the examiner to consider the applicability of 35 U.S.C. 103(c) and potential 35 U.S.C. 102(e), (f) or (g) prior art under 35 U.S.C. 103(a).

11. Claims 1-8, and 11-20 are rejected under 35 U.S.C. 103(a) as being unpatentable over Gravley (US 4765964), or alternatively over Gravley in view of Voll et al., "Carbon Black" in: *Ullmann's Encyclopedia of Industrial Chemistry* (1986 ed.), vol. A5, pp. 144-148.

12. With respect to claim 1, Gravley discloses a process for carrying out a high-temperature reaction, in which starting materials are supplied to a reaction chamber through channels of a burner block (see Gravley, column 3, lines 15-16), where in the reaction chamber the high-temperature reaction having a short residence time takes

place at a temperature of at least 1500°C (see Gravley, column 7, lines 56-60) and the reaction mixture is subsequently rapidly cooled in the quench area (see Gravley, column 6, lines 37-39), characterized in that in the quench area firstly a direct cooling takes place by supply of an evaporating quench medium.

Gravley does not disclose wherein the direct cooling results in a lowering of the temperature to the range from 650°C to 1200°C nor that such direct cooling is followed by indirect cooling in a heat exchanger.

However, such process conditions are known in the art for the same type of process that Gravley discloses. For example, Voll discloses a process whereby direct cooling results in a lowering of the temperature to 800°C (see Voll, page 146) followed by indirect cooling in a heat exchanger (see Voll, page 147).

Therefore, it would have been obvious to the person having ordinary skill in the art at the time the invention was made to operate the process of Gravley at conditions whereby indirect cooling resulted in a lowering of the temperature to the range from 650°C to 1200°C followed by indirect cooling in a heat exchanger.

13. With respect to claim 2, Gravley discloses wherein the starting materials are premixed (see Gravley, column 3, lines 24-27).

14. With respect to claim 3, Voll discloses direct cooling resulting in a lowering of the temperature to 800°C (see Voll, page 147).

15. With respect to claim 4, Gravley discloses wherein direct cooling takes place in one stage (see Gravley, column 6, lines 37-43).

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16. With respect to claim 5, Gravley discloses wherein the quench medium is water (see Gravley, column 6, lines 51-54).

17. With respect to claim 6, Voll discloses indirect cooling to a temperature less than 300°C (see Voll, page 147).

18. With respect to claim 7, Voll discloses wherein the indirect cooling is utilized for the generation of steam (see Voll, page 147).

19. With respect to claim 8, Gravley discloses wherein all surfaces restricting the reaction chamber are formed of a fire-resistant ceramic having an alumina content of at least 80% by weight (see Gravley, column 5, lines 49-53).

20. With respect to claims 11 and 12, Gravley discloses a transition of the reaction chamber to quench area designed in the form of an annular gap having a width in the range from 2 to 200 mm (see Gravley, column 6, lines 31-34, and column 10, line 39).

21. With respect to claim 13, Gravley discloses a reaction chamber designed in the form of an annular gap (see Gravley, Figure).

22. With respect to claims 14 and 15, Gravley discloses channels in the burner block aligned in the direction of the longitudinal axis of the reaction chamber (see Gravley, Figure).

23. With respect to claim 16, Gravley discloses a quench area constructed with alignment in the direction of the longitudinal axis of the reaction chamber (see Gravley, Figure).

24. With respect to claims 17 and 18, Gravley discloses the supply of quench medium via quench nozzles attached to one or more distributors arranged radially to the main flow direction of the reaction mixture (see Gravley, Figure).

25. With respect to claim 19, Gravley discloses a process for the scale-up of a reactor characterized in that for a throughput enlargement the internal diameter of the reactor is enlarged and the gap size at the transition from the reaction chamber to the quench area is kept constant (see Gravley, Table I, runs 8 and 9).

26. With respect to claim 20, acetylene is a known product of the partial combustion of methane with oxygen. Thus, Gravley provides an inherent disclosure for a method for the preparation of acetylene by partial oxidation of hydrocarbons using oxygen.

27. Claims 9 and 10 are rejected under 35 U.S.C. 103(a) as being unpatentable over Gravley (US 4765964) in view of Bakker (US 3640739).

28. With respect to claim 9, Gravley discloses a process for carrying out a high-temperature reaction, in which starting materials are supplied to a reaction chamber through channels of a burner block (see Gravley, column 3, lines 15-16), where in the reaction chamber the high-temperature reaction having a short residence time takes place at a temperature of at least 1500°C (see Gravley, column 7, lines 56-60) and the reaction mixture is subsequently rapidly cooled in the quench area (see Gravley, column 6, lines 37-39), characterized in that in the quench area firstly a direct cooling takes place by supply of an evaporating quench medium, and characterized in that all surfaces restricting the reaction chamber are formed of a fire-resistant ceramic having an alumina content of at least 80% by weight (see Gravley, column 5, lines 49-53).



Gravley does not disclose wherein the fire-resistant ceramic is introduced into the reaction chamber in the form of stones or bricks or as a cast or tamped mass and subsequently compressed, dried, and calcined.

However, Bakker discloses a refractory material made from a high purity alumina refractory brick batch mix consisting of 85% – 95% alumina by weight (see Bakker, column 2, lines 10-12). Bakker discloses that the refractories of his invention are of increased strength, higher density, lower porosity, and higher refractoriness than other refractories commercially available (see Bakker, column 1, lines 62-67). Bakker further discloses whereby the alumina refractory ("fire-resistant ceramic") is shaped into bricks, compressed, dried, and calcined (see Bakker, column 3, lines 58-70).

Therefore, it would have been obvious to the person having ordinary skill in the art at the time the invention was made to line the inside of the reaction chamber of Gravley with the fire-resistant alumina refractory of Bakker so as to provide a more durable refractory sufficient for use under high reaction temperatures.

29. With respect to claim 10, Bakker discloses pressing the refractory mix into any desired shape (see Bakker, column 3, lines 58-59).

### ***Conclusion***

30. Any inquiry concerning this communication or earlier communications from the examiner should be directed to Randy Boyer whose telephone number is (571) 272-7113. The examiner can normally be reached Monday through Friday from 8:00 A.M. to 5:00 P.M.

Art Unit: 1764

If attempts to reach the examiner by telephone are unsuccessful, the examiner's supervisor, Glenn A. Caldarola, can be reached at (571) 272-1444. The fax number for the organization where this application or proceeding is assigned is 571-273-8300.

Information regarding the status of an application may be obtained from the Patent Application Information Retrieval (PAIR) system. Status information for published applications may be obtained from either Private PAIR or Public PAIR. Status information for unpublished applications is available through Private PAIR only. For more information about the PAIR system, see <http://pair-direct.uspto.gov>. Should you have questions on access to the Private PAIR system, contact the Electronic Business Center (EBC) at 866-217-9197 (toll-free). If you would like assistance from a USPTO Customer Service Representative or access to the automated information system, call 800-786-9199 (IN USA OR CANADA) or 571-272-1000.

RPB



Glenn A. Caldarola  
Supervisory Patent Examiner  
Technology Center 1700

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Sheet 1 of 1Document Number  
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Application Number

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IN AN APPLICATION

Applicant Bartenbacg et ak,

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(Use several sheets if necessary)

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	44 22815	1/96	Germany			
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OTHER DOCUMENTS (Including Author, Title, Date, Pertinent Pages, Etc.)						
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KEIL &amp; WEINKAUF

**Notice of References Cited**

Application/Control No.

10/806,191

Applicant(s)/Patent Under  
Reexamination  
BARTENBACH ET AL.

Examiner

Randy Boyer

Art Unit

1764

Page 1 of 1

**U.S. PATENT DOCUMENTS**

*		Document Number Country Code-Number-Kind Code	Date MM-YYYY	Name	Classification
*	A	US-4,765,964	08-1988	Gravley et al.	422/156
*	B	US-3,640,739	02-1972	Bakker, Wate T.	501/129
	C	US-			
	D	US-			
	E	US-			
	F	US-			
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	N					
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	P					
	Q					
	R					
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**NON-PATENT DOCUMENTS**

*		Include as applicable: Author, Title Date, Publisher, Edition or Volume, Pertinent Pages)
	U	Voll et al., "Carbon Black" in: Ullmann's Encyclopedia of Industrial Chemistry (1986 ed.), vol. A5, pp. 144-148.
	V	
	W	
	X	

\*A copy of this reference is not being furnished with this Office action. (See MPEP § 707.05(a).)  
Dates in MM-YYYY format are publication dates. Classifications may be US or foreign.

# Ullmann's Encyclopedia of Industrial Chemistry

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Editors: F. Thomas Campbell, Rudolf Pfefferkorn,  
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## Carbon

OTTO VOHLER, FERDINAND VON STURM, ERHARD WEGE, Sigr GmbH, Meitingen, Federal Republic of Germany (Chaps. 1-3)

HARTMUT VON KIENTLE, Degussa AG, Zweigniederlassung Wolfgang, Hanau, Federal Republic of Germany (Chap. 4)

MANFRED VOLL, PETER KLEINSCHMIT, Degussa AG, Zweigniederlassung Wolfgang, Hanau, Federal Republic of Germany (Chap. 5)

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Acetylene, due to its high price, is used only for the production of highly specialized conductivity blacks and battery blacks.

#### 5.4. Production Processes

A summary of the most important production processes is given in Table 16. In general, the processes are divided into two groups: those employing partial combustion and those based on pure pyrolysis. This nomenclature is somewhat misleading insofar as the carbon black resulting from the partial combustion process is also formed by pyrolysis. The two types of processes differ in that air is used in the one to burn part of the feedstock, thus producing the energy required to carry out the pyrolysis, whereas in the other heat is generated externally and introduced into the process.

The furnace black process is currently the most important production process. It accounts for more than 95% of the total worldwide production. The advantages of the furnace black process are its great flexibility in manufacturing various grades of carbon black and its better economy. The following comparison makes this apparent: for similar grades of carbon black, the production rate of one flame is ca. 0.002 kg/h for channel black, ca. 0.2 kg/h for gas black, and ca. 2000 kg/h for a modern furnace black reactor. However, in spite of the more advantageous furnace black process, the production processes listed in Table 16 (except for the channel black process) are still in use for the production of special carbon blacks which cannot be obtained via the furnace black process.

Table 16. Summary of the manufacturing processes and feedstocks for the production of carbon black

Chemical process	Manufacturing process	Feedstock
Incomplete combustion	furnace black process	petrochemical oils and coal tar oils
	Degussa gas black process	coal tar oils
	channel black process	natural gas
	lamp black process	petrochemical and coal tar oils
Thermal cracking	thermal black process	natural gas
	acetylene black process	acetylene
	Hüls arc process (carbon black as byproduct in acetylene production)	light hydrocarbons

##### 5.4.1. Furnace Black Process

The rapidly expanding automobile industry required increasing numbers of tires with various characteristics. This led not only to the development of new rubber grades, but also to the development of new carbon blacks required by the increasingly refined application processes and to the development of a new and better manufacturing process, the furnace black process. Unlike the old channel black process, this process allows the production of nearly all types of carbon black required by the rubber industry. It also meets the high economic and ecological requirements of our times.

The furnace black process was developed in the United States in the 1920s, and since then, it has been greatly refined. It is a continuous process, carried out in closed reactors, so that all inputs can be carefully controlled [282]. Today most semireinforcing rubber blacks of the types SRF, APF, GPF, and FEF with specific surface areas of 20–60 m<sup>2</sup>/g and the active reinforcing blacks of the types HAF, ISAF, and SAF (see Table 19, p. 155) with specific surface areas of 65–150 m<sup>2</sup>/g are manufactured by this process, as well as to an increasing extent, pigment-grade carbon blacks with much greater specific surface areas and smaller particle sizes. In addition to the specific surface area, other quality specifications such as structure, measured as DBP absorption, and application properties of rubber such as abrasion resistance, modulus, and tear strength or jetness and tinting strength for color blacks can also be systematically varied in the furnace black process by adjusting the operating parameters. This flexibility is necessary to meet the very narrow specifications required by customers.

The heart of a furnace black production plant is the furnace in which the carbon black is formed. The feedstock is injected, usually as an atomized spray, into a high-temperature and high-energy density zone, which is achieved by burning a fuel (natural gas or oil) with air. The oxygen, which is in excess with respect to the fuel, is not sufficient for the complete combustion of the feedstock, which, therefore is for the most part pyrolyzed to form carbon black at temperatures of 1200–1900 °C. After the reaction mixture is quenched with water and further cooled in heat exchangers, the carbon black is collected from the tail gas by using a filter system.

Figure 34 shows a schematic drawing of a furnace black plant. The feedstock, preferably petrochemical or carbochemical heavy oils, which usually begin to crystallize above the ambient temperature, are stored in heated tanks equipped with circulation pumps to maintain a homogeneous mixture. Oil is conducted to the reactor by means of rotary pumps via heated pipes and a heat exchanger, where it is heated to 150–250 °C to obtain a viscosity appropriate for atomization. Various types of spraying devices are used to introduce the feedstock into the reaction zone. An axial oil injector with a spraying nozzle at its tip, which usually produces a hollow-cone spray pattern, is a commonly used device. One- and two-component atomizing nozzles [283] are in use, air and steam being the preferred atomizing agents in the latter case. However, the feedstock is injected into some reactors as a plurality of coherent streams into the accelerated combustion gases perpendicular to the direction of stream [284].

As the carbon black structure may be reduced by the presence of alkali metal ions in the reaction zone [285], alkali metal salts, preferably aqueous solutions of potassium hydroxide or potassium chloride, are often added to the make oil in the oil injector. Alternatively, the additives may be sprayed separately into the combustion chamber. In special cases, other additives, e.g., alkaline-earth metal compounds which increase the specific surface area are introduced in a similar manner.

The high temperature necessary for pyrolysis is obtained by burning fuel in excess air in a combustion chamber. Natural gas is still the fuel of choice, but other gases, e.g., coke oven gases or vaporized liquid gas, are occasionally used. Various oils including the feedstock will probably be used as fuel for economic reasons in the future. Recycled off-gas (tail gas) processes in combination with oxygen-enriched air have recently been proposed. Special burners, depending on the type of fuel, are used to obtain fast and complete combustion.

The air required for combustion is compressed by rotating piston compressors or turbo blowers. The air is preheated in heat exchangers by the hot gases containing carbon black which are leaving the reactor, which conserves energy and thus improves the carbon black yield. Temperatures of 300–600 °C for preheating are normal, but there is a tendency to further increase the temperature.

Important progress has been made on the reactor throughput: A production plant with a capacity of 20 000 t/a (2.5 t/h) was previously run with as many as 12 furnaces, which in the last decade (in most cases) have been replaced by only one high-performance reactor. Modern plants are one-stream units with only one aggregate for each process step (reactor, collecting system, beading device, dryer). From a commercial point of view, even larger units are possible. However, due to the great variety of carbon black types required, the capacity of one unit is

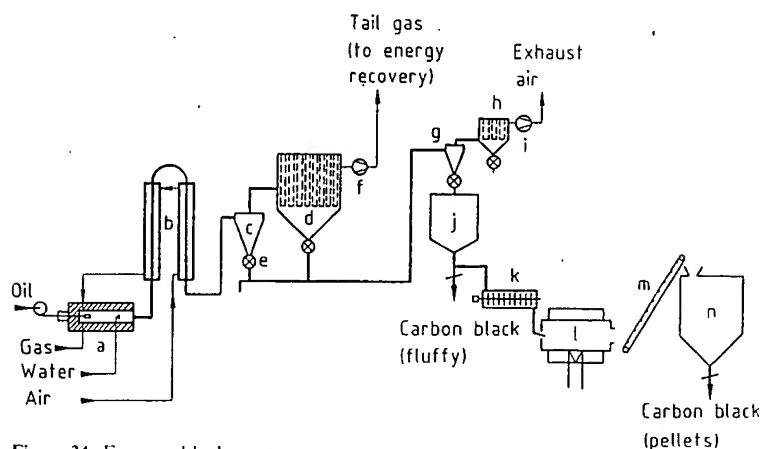


Figure 34. Furnace black process

a) Furnace black reactor; b) Heat exchanger; c) Collecting or agglomerating cyclone; d) Bag filter; e) Carbon black outlet to pneumatic conveying system; f) Tail gas blower; g) Collector; h) Exhaust air filter; i) Blower for the pneumatic conveying air; j) Fluffy black storage tank; k) Beading machine; l) Dryer drum; m) Conveying belt; n) Storage tank for carbon black beads

economically limited by the frequency of switching over to other types and the amount of off-grade carbon black produced during this procedure.

The reactors of modern furnace plants vary considerably in shape, flow characteristics, and the manner in which fuel and feedstock are introduced. Nevertheless, they all have the same basic process steps in common: producing hot combustion gases in a combustion chamber, injecting the feedstock and rapidly mixing it with the combustion gases, vaporizing the oil, pyrolyzing it in the reaction zone, and rapidly cooling the reaction mixture in the quenching zone to temperatures of 500–800 °C.

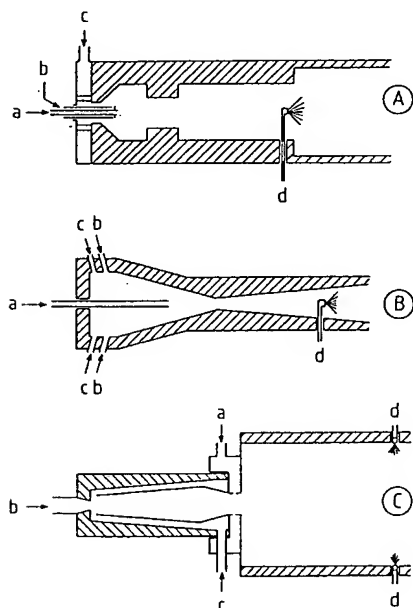
Schematic drawings of some typical modern furnace black reactors are shown in Figure 35. They all have a gas-tight metal jacket. The reaction zone is coated with a ceramic inner liner, generally on an alumina base, which is stable to temperatures of ca. 1800 °C. Several quenching positions allow the changing of the effective volume of the reactor. This allows variation of the mean residence time of the carbon black at the high reaction temperature. Typical residence times for reinforcing blacks are 10–100 ms.

Most furnace black reactors are arranged horizontally. They can be up to 18 m long with

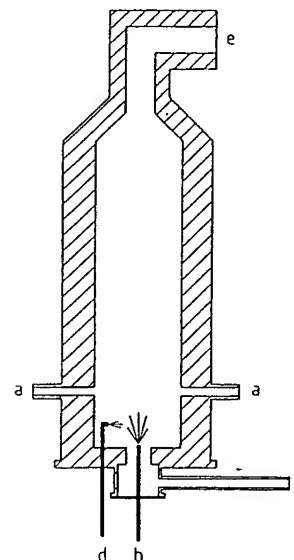
an outer diameter of up to 2 m. Some vertical reactors are used especially for the manufacture of certain semireinforcing blacks [288] (Fig. 36). For further reactors, see [289].

The properties of carbon blacks are dependent on, e.g., the ratios of fuel, feedstock, and air, which therefore must be controlled carefully [290]. The particle size of the carbon black formed generally decreases with increasing amounts of excess air relative to the amount needed for the complete combustion of the fuel. Since the excess air reacts with the feedstock, a greater amount of air leads to higher oil combustion rates, resulting in rising temperatures in the reaction zone. As a consequence, the nucleation velocity and the number of particles formed increase, but the mass of each particle and the total yield decrease. This allows semireinforcing carbon blacks to be manufactured with better yields than active reinforcing carbon blacks. The yields, which depend on the carbon black type and the type of feedstock, range between 50 and 65% for semireinforcing blacks and 40 and 60% for reinforcing blacks. High-surface-area pigment black with markedly smaller particle size than rubber blacks gives lower yields.

Other parameters influencing carbon black quality are the manner in which the oil is in-



**Figure 35.** Furnace black reactors  
A) Restrictor ring reactor; B) Venturi reactor [286]; C) Reactor with high-speed combustion chamber [287]  
a) Feedstock; b) Fuel; c) Combustion air; d) Quench



**Figure 36.** Vertical reactor for manufacturing semireinforcing blacks  
a) Fuel inlet; b) Oil injector; c) Air conduit; d) Water spray; e) Outlet to the collecting system

jected, atomized, and mixed with the combustion gases, the type and amount of additives, the preheating temperature of the air, and the quench position. As long as the carbon black is in contact with the surrounding gases at the high reaction temperature, several reactions on the carbon surface occur (e.g., Boudouard reaction, water gas reaction), so that the chemical nature of the carbon black surface is modified with increasing residence time. When quenched to temperatures  $< 900^{\circ}\text{C}$ , these reactions are stopped and a certain state of surface activity is frozen. Carbon black surface properties can also be adjusted by varying the beading and drying conditions (see below).

Typical processing data for reactors with a carbon black output of 10 000 t/a (1250 kg/h) of tread black and of 14 000 t/a (1750 kg/h) of carcass black are listed in Table 17. These data show that the total mass put through the reactor amounts to 10–16 t/h. Although this is done at high streaming velocities (up to 800 m/s) and high temperatures (up to  $1800^{\circ}\text{C}$ ), modern high-performance reactors have lifetimes of 2 years and more.

The mixture of gas and carbon black leaving the reactor is cooled to temperatures of  $250\text{--}350^{\circ}\text{C}$  in heat exchangers by counterflowing combustion air and then conducted into the collecting system. Formerly, a combination of electroflocculators and cyclones or cyclones and filters were used [282], [291]. Currently, simpler units are preferred. Generally, the collecting system consists of only one high-performance bag filter with several chambers, which are periodically purged by counterflowing filtered gas. Occasionally, an agglomeration cyclone is installed between the heat exchanger and the filter. Depending on the capacity of the production unit, the filter may contain several hundred bags with a total filter area of several thousand square meters. Usual filter loads are on the order of  $0.2\text{--}0.4\text{ m}^3\text{ m}^{-2}\text{ min}^{-1}$ . Since the filtered gas contains 25–40 vol% water vapor, most filters

operate at temperatures above  $200^{\circ}\text{C}$  to avoid condensation. The residual carbon black content in the off-gas is less than  $50\text{ mg/m}^3$ .

Because of the reducing atmosphere and the high temperatures in the reactor, the tail gas, which consists of 25–40 vol% water vapor, 40–50 vol% nitrogen, and 3–5 vol% carbon dioxide, also contains a certain amount of combustible gases, the amount of which depends on the feedstock and the processing conditions. These gases include 5–10 vol% carbon monoxide, 5–10 vol% hydrogen, and small amounts of methane and other hydrocarbons. The lower heating value lies between 1700 and  $2100\text{ kJ/m}^3$ . The energy remaining in the tail gas can be calculated by using the typical overall energy balance of the furnace black process shown in Figure 37. The gas, which must be burned off for environmental reasons, is used as energy, e.g., for heating dryer drums or for the production of steam and electricity.

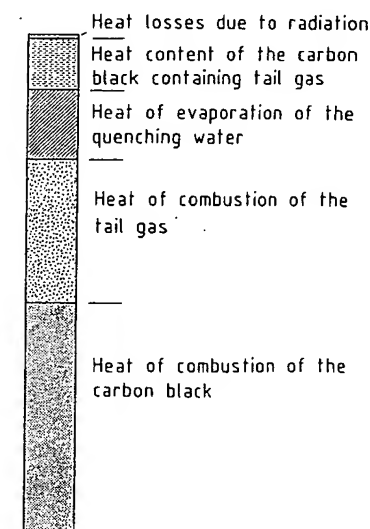


Figure 37. Typical energy balance for the manufacture of a HAF black

Table 17. Processing data for high-performance furnace black reactors

		Semireinforcing carbon black (FEF)	Reinforcing carbon black (ISAF)
Natural gas	$\text{m}^3/\text{h}$	300–550	280–440
Air	$\text{m}^3/\text{h}$	7000–10 000	6000–7500
Oil	$\text{kg/h}$	2500–3300	2000–3000
Carbon black	$\text{kg/h}$	1500–2000	1000–1500
	$\text{t/a}$	12 000–16 000	8000–12 000
Yield	$\text{kg/100 kg of oil}$	50–65	40–55

The fluffy carbon black coming out of the filter is pneumatically conveyed into a first storage tank. Small amounts of impurities ("grit," e.g., iron, rust, or coke particles) are either removed by magnets and classifiers or milled to an appropriate consistency.

Freshly collected carbon black has an extremely low bulk density of 20–60 g/L. To facilitate handling and further processing by the customer, it must be compacted. "Outgassing," a process by which the carbon black is conducted over porous, evacuated drums, is the weakest form of compacting which allows the carbon black to retain its powdery state [292]. This form of compacting is used for certain pigment blacks which must remain very dispersible.

Other pigment blacks and the rubber blacks are compacted by beading. Two pelletizing processes are used: dry and wet beading. Dry beading is a simple and energy-saving method, but it does not work with all types of carbon black. It is mainly used for color blacks. Dry beading is carried out in rotating drums, where the powdery carbon black rolls to form small spheres.

The majority of rubber blacks is pelletized by the wet-beading process. Carbon black, water, and small amounts of beading additives, e.g., molasses, ligninosulfonic acids, are mixed in special beading machines [293]. They usually consist of a horizontal, cylindrical trough ca. 3 m long and 0.7–1 m in diameter in the axis of which a pin shaft rotates at 300–750 rpm (Fig. 38). The pins are positioned helically around the shaft. The water and the beading additives dissolved in it are injected via spray nozzles. The density is ca. 10 times that of the original carbon black. DBP absorption is also reduced during this process. The pellet strength and some application properties in rubber can be influenced by the type and amount of the beading additives. The size of the pellets is ca. 1 mm.

The carbon black leaving the beading machine contains ca. 50 wt% water. It is dried in

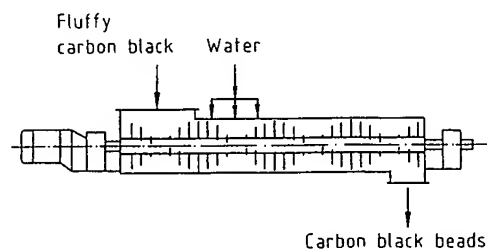


Figure 38. Beading machine

indirectly heated dryer drums, e.g., by burning tail gas. Dryer drums with a capacity of 2000 kg of carbon black per hour are 15–20 m long and 2–3 m in diameter. They are rotated at 5–15 rpm. Drying temperatures, generally between 150 and 250 °C, allow further modification of the carbon black properties.

The dried carbon black is transported via conveyor belts and elevators to the storage tank or packing station. Bulk densities of wet-beaded carbon blacks are between 250 and 500 g/L.

A flow diagram summarizing the complete furnace black process is shown in Figure 39.

#### 5.4.2. Gas Black and Channel Black Processes

The channel black process, used in the United States since the late 19th century, is the oldest process for producing small-particle-size carbon blacks on an industrial scale. Originally, the first reinforcing blacks were also produced by this process. In 1961, the production of channel black was about 120 000 t. The last production plant in the United States was closed in 1976, due to low profitability and environmental difficulties. Natural gas was used as the feedstock. The carbon black yield was only 3–6%.

In Germany, where natural gas was not available in sufficient amounts, the gas black process was developed in the 1930s. It is similar to the channel black process, but uses coal tar oils instead of natural gas. Yields and production rates are much higher with oil-based feedstock; this process is still used to manufacture high-quality pigment blacks with properties comparable to those of channel blacks. The gas black process has been used by Degussa on an industrial scale since 1935.

Originally, gas black was primarily used for the reinforcement of rubber. Only one special gas black is still used in the rubber industry today; all other types are used as color blacks in printing inks, plastics, lacquers, and coatings. High-quality oxidized gas blacks (see p. 152) are of special interest, e.g., in deep black lacquers and coatings.

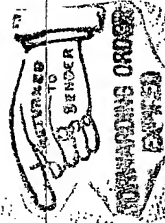
In the gas black process (Fig. 40), the feedstock (coal tar oil, raw naphthalene, or anthracene oil) is partially vaporized. The residual oil is continuously withdrawn. The oil vapor is transported to the production apparatus by a combustible carrier gas (e.g., hydrogen, coke oven gas, or methane). Air is added to the

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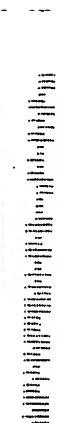
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